

## CLAIMS

1. A process for producing a poly(arylene sulfide), comprising, after a dehydration step of heating and  
5 dehydrating a mixture containing an organic amide solvent, at least one sulfur source (A) selected from the group consisting of alkali metal hydrosulfides and alkali metal sulfides and an alkali metal hydroxide added as needed to control the amount of water in the mixture, a  
10 polymerization step of charging a dihalo-aromatic compound (B) into the system containing the remaining mixture to subject the sulfur source (A) and the dihalo-aromatic compound (B) to a polymerization reaction in the organic amide solvent, which comprises:
- 15 (1) in the dehydration step, heating the mixture containing the organic amide solvent, at least one sulfur source (A) selected from the group consisting of alkali metal hydrosulfides and alkali metal sulfides and the alkali metal hydroxide added as needed in a reaction vessel, to  
20 which a distillation column is linked, and guiding vapor volatilized to the distillation column to distill and separate it into respective components,
- refluxing a fraction taken out of the bottom of the distillation column and comprising the organic amide  
25 solvent as a principal component into the reaction vessel,
- cooling a fraction taken out of the top of the distillation column and comprising water and hydrogen

sulfide to discharge hydrogen sulfide that is not condensed by the cooling and reflux a part of water condensed into the distillation column, discharging the remaining water, (2) determining a relational expression between the total amount of water of an amount of water refluxed into the distillation column and an amount of water discharged without being refluxed, and an amount of hydrogen sulfide discharged from the reaction vessel in advance, thereby calculating out an amount of hydrogen sulfide discharged from the reaction vessel from a measured value of the total amount of water on the basis of the relational expression, (3) calculating out an amount (hereinafter referred to as "amount of the sulfur source charged") of the sulfur source (A) remaining in the mixture after the dehydration step on the basis of the amount of hydrogen sulfide calculated out, thereby controlling a charged molar ratio of the sulfur source (A) to the dihalo-aromatic compound (B) on the basis of the amount of the sulfur source (A) calculated out, and then (4) subjecting the sulfur source (A) and the dihalo-aromatic compound (B) to the polymerization reaction in the organic amide solvent in the polymerization step.

2. The production process according to claim 1, wherein the relational expression is a linear relational expression represented by the following relational expression (I):

$$y = ax + b \quad (I)$$

wherein x is the total amount of the amount of water  
refluxed into distillation column and the amount of water  
discharged without being refluxed in the dehydration step,  
5 y is the amount of hydrogen sulfide discharged from the  
reaction vessel, and both a and b are parameters.

3. The production process according to claim 1,  
wherein in the dehydration step, the mixture is heated to a  
10 temperature of 100 to 250°C.

4. The production process according to claim 1,  
wherein in the dehydration step, reflux conditions are set  
in such a manner that a weight ratio of the amount of water  
15 refluxed into the distillation column to the amount of  
water discharged without being refluxed falls within a  
range of from 90:10 to 10:90.

5. The production process according to claim 1,  
20 wherein in the dehydration step, the dehydration under heat  
is conducted in such a manner that the water content falls  
within a range of 0.3 to 5 mol per mol of the alkali metal  
sulfide (A) charged.

25 6. The production process according to claim 1,  
wherein in the dehydration step, the dehydration under heat  
is conducted by means of an apparatus so constructed that

an upper part of the reaction vessel is connected to the distillation column, a fraction from the top of the distillation column is successively sent to a condenser and a storage tank, a fraction from the bottom of the distillation column is refluxed into the reaction vessel, a part of water stored in the storage tank is refluxed into the distillation column, and at that time an amount of water refluxed is integrated by a flowmeter.

10           7. The production process according to claim 1, wherein after the dehydration step, an amount of the dihalo-aromatic compound (B) charged is controlled within a range of 1.00 to 1.09 mol per mol of the sulfur source (A) charged.

15           8. The production process according to claim 1, wherein in the polymerization step, the polymerization reaction is conducted by an at least two-stage polymerization process comprising:

20   (1) Step 1 of heating a reaction mixture containing the organic amide solvent, the sulfur source (A) and the dihalo-aromatic compound (B) to 170 to 270°C in the presence of water in an amount of 0.5 to 2.0 mol per mol of the sulfur source (A) charged to conduct a polymerization  
25 reaction, thereby forming a prepolymer that a conversion of the dihalo-aromatic compound is 50 to 98%, and  
(2) Step 2 of controlling the amount of water in the

reaction system so as to bring about a state that water exists in a proportion of more than 2.0 mol, but up to 10 mol per mol of the sulfur source (A) charged, and heating the reaction system to 245 to 290°C, thereby continuing the polymerization reaction.

9. The production process according to claim 8, wherein in Step 1, a prepolymer having a melt viscosity of 0.5 to 30 Pa·s as measured at a temperature of 310°C and a shear rate of 1,216 sec<sup>-1</sup> is formed.